

## PATENT ABSTRACTS OF JAPAN

English abstract and  
English excerpted  
translations of  
Document 1)

(11)Publication number : 06-306355

(43)Date of publication of application : 01.11.1994

(51)Int.Cl.

C09K 11/00

C09K 11/08

C09K 11/56

H05B 33/14

(21)Application number : 05-100579

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(22)Date of filing : 27.04.1993

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## (54) PRODUCTION OF PHOSPHOR

## (57)Abstract:

**PURPOSE:** To produce a phosphor used for an electroluminescent lamp and having a high luminance and a long life by baking a mixture contg. zinc sulfide, a copper compd., and a halogen compd. at a relatively high temp., applying impact to the resulting intermediate phosphor to cause strain in its particles, and baking the intermediate phosphor at a temp. lower than the above baking temp.

**CONSTITUTION:** 100 pts.wt. finely powdered zinc sulfide is mixed and stirred with a copper compd. (e.g. copper sulfate) in an amt. of Cu of 0.05-0.2mol% together with pure water. The mixture is dried under heating in an oven, mixed with 5-25mol% flux comprising an alkaline earth metal halide and at least two alkali metals, and baked at 900-1,100° C for 1-5hr in air. The resulting intermediate phosphor is put into a container together with balls having diameters of 0.1-10mm and stirred under 100-1,500rpm for 10-240min to apply impact to the phosphor. The phosphor with impact strain in its particles is baked at 500-800° C for 15hr in air. Then the surface of the phosphor is washed with an aq. KCN soln. and water and dried.

## LEGAL STATUS

[Date of request for examination] 26.01.1995

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number] 2696809

[Date of registration] 19.09.1997

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right] 19.09.2002

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**CLAIMS**

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**[Claim(s)]**

[Claim 1] The manufacture approach of the fluorescent substance characterized by including the process which calcinates comparatively the mixture which added the copper compound and the halogenated compound to zinc sulfide at an elevated temperature, and manufactures a medium fluorescent substance, the process which impulse force is applied [ process ] to said medium fluorescent substance, and produces distortion in a fluorescent substance particle, and the process which the medium fluorescent substance which produced said distortion is calcinated [ process ] at low temperature rather than said baking, and makes copper segregate.

[Claim 2] The manufacture approach of the fluorescent substance according to claim 1 characterized by for the process which impulse force is applied [ process ] to said medium fluorescent substance particle, and produces distortion in a particle making a medium fluorescent substance particle and a sphere collide, and being in the range whose spherical diameter of this is 0.1-5mm.

[Claim 3] The manufacture approach of the fluorescent substance according to claim 2 characterized by said spherical specific gravity becoming the range of 2.0-6.0.

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] Especially this invention relates to the manufacture approach of an electroluminescence fluorescent substance about the manufacture approach of a fluorescent substance.

[0002]

[Description of the Prior Art] An electroluminescence LGT is used for the back light of quiescence displays, such as an alphabetic character and a graphic form, or a liquid crystal display etc., and explains the structure with reference to drawing 2. Through the moisture absorption films 2 and 2, with the envelope films 3 and 3 of two more sheets, a sandwiches type pinches the front flesh side of electroluminescence LGT 10 of the electroluminescence devices 1 of a flat rectangle, and it closes it. Electroluminescence devices 1 are what carried out the laminating of the back plate 4 which consists of aluminum foil, the reflective insulating layer 5, the luminous layer 6 containing a fluorescent substance, and the transparent electrode 7 from the rear-face side to the front-face side, and the leads 8 and 8 for electrode cash drawers of two which consist of one side of the above-mentioned back plate and a transparent electrode are drawn. You distribute much minute spherical fluorescent substance 6b, and the above-mentioned luminous layer 6 makes it mix in organic binder 6a, as shown in drawing 3. If high tension is impressed between a back plate 4 and a transparent electrode 7 from the above-mentioned leads 8 and 8, fluorescent substance 6b of two electrodes 4 and the luminous layer 6 between seven will emit light by two electrodes 4 and the electric field formed among seven.

[0003] Generally, fine-particles-like zinc sulfide (ZnS) is used as a parent, and the above-mentioned fluorescent substance 6b is obtained by calcinating the mixture which added the halogen (for example, Cl) as copper (Cu) and co-activating agent as an activator to the parent.

[0004] This manufacture approach is explained. the inside of the group which becomes this mixture from the halogenide and alkali metal of alkaline earth metal as a fusing agent after adding a 0.02 - 0.1wt% copper sulfate, making it the shape of a slurry, mixing and drying to the zinc sulfide 100 weight section — at least one kind — 5 - 10wt% — it mixes. The appropriate back, this mixture is put into a quartz crucible, and it covers, and calcinates in 3 - 10-hour air at the temperature of the range of 1100-1200 degrees C, and by the deionized water after baking, it washes several times, it dries, and a medium fluorescent substance is made. High voltage is applied to this medium fluorescent substance, and further, if it anneals at 700-950 degrees C, the fluorescent substance for electroluminescence LGTs can be manufactured. (For example, JP,61-296085,A)

[Problem(s) to be Solved by the Invention] By the way, the manufacture approach of the above-mentioned conventional fluorescent substance for electroluminescence LGTs is what was pressurized with hydrostatic pressure in order to transfer the crystal mold of a fluorescent substance, and it had the fault that formation of the conductive layer (called CuX S) by the copper segregation was not enough, and brightness and lives were insufficient.

[0005] This invention aims at offering the manufacture approach of the fluorescent substance for electroluminescence LGTs of having attained high brightness and reinforcement by forming the conductive layer which forms distortion in a fluorescent substance particle and originates in this distortion.

[0006]

[Means for Solving the Problem] In order to attain the above-mentioned object, the manufacture approach of the fluorescent substance of this invention The process which calcinates comparatively the mixture which added the halogenated compound used as the copper compound used as an activator, and co-

activating agent to zinc sulfide at an elevated temperature, and manufactures a medium fluorescent substance. It is characterized by including the process which applies impulse force to said medium fluorescent substance, is distorted in a fluorescent substance particle, and forms \*\*, and the process which manufactures the fluorescent substance which calcinated the medium fluorescent substance which produced said distortion at low temperature rather than said baking, and was made to segregate copper. [0007] Moreover, in the process which impulse force is applied [ process ] to a medium fluorescent substance by the collision of a sphere and a medium fluorescent substance particle, and produces distortion in a particle, it is characterized by for a path being in a sphere and 0.1–5mm and the specific gravity of those being in the range of 2.0–6.0.

[0008]

[Function] By the above spheres, if impulse force is applied to a medium fluorescent substance particle, distortion can be produced in a medium fluorescent substance particle, without giving stress to the extent that a medium fluorescent substance particle is broken. Without reducing EL property, since a medium fluorescent substance particle is not broken, since especially particle size does not become small, effect good for a life property is brought about.

[0009] Since distortion is produced in the medium fluorescent substance particle, a copper segregation is comparatively produced easily by low-temperature baking, and since this copper that segregated works as a conductive layer, brightness improves substantially.

[0010] Although a medium fluorescent substance particle is hard, it has a weak property. Therefore, when a fluorescent substance particle is pressurized, it will return, if it is strong to compressive stress and application of pressure is removed, and is hard to make a fluorescent substance particle produce distortion with an isotropic pressure like hydrostatic pressure. However, since a pressure is applied in different direction, a fluorescent substance particle can be made to produce distortion in the stress by the collision of a sphere like this invention, and a fluorescent substance particle.

[0011]

[Example] Hereafter, this invention is explained based on an example.

[0012] After adding a copper compound, for example, a copper sulfate, to the impalpable powder-like zinc sulfide 100 weight section as usual so that Cu concentration may become 0.05–0.2-mol%, mixing with pure water and stirring well, stoving of this mixture is carried out within oven. the inside of the group which becomes the dry mixture from the halogenide and alkali metal of alkaline earth metal as flux — at least two or more sorts — total amount % of 5–25 mols — it mixes.

[0013] next, this mixture — an alumina crucible — putting in — a cover — carrying out — the range of 900–1100 degrees C — it calcinates in 1 – 5-hour air at an elevated temperature comparatively. Then, impalpable powder-like zinc sulfide condenses through flux and it grows up to be a 10–40-micrometer particle. The medium fluorescent substance and a ball with a diameter of 0.1–10mm were put into the container, it stirred for 10 – 240 minutes at the rotational frequency of 100 – 1500rpm, and impulse force was applied to the medium fluorescent substance. On this condition, distortion can be formed in a particle, without breaking a medium fluorescent substance particle or grinding.

[0014] Next, the medium fluorescent substance which produced the distortion is comparatively calcinated rather than the above mentioned baking in 500–800 degrees C of low temperature, and 1 – 5-hour air. the copper which was carrying out homogeneity diffusion into the medium fluorescent substance particle by said baking carried out then was heated — moving — being easy — copper is segregated and a conductive layer is formed in a location with the distortion in a particle.

[0015] Then, the KCN water solution removed the discard in a fluorescent substance front face as usual. After carrying out backwashing by water of the KCN component, within oven, it dries, and it sifts out and completes as a fluorescent substance.

[0016] Thus, if the produced fluorescent substance 20 is distributed in organic binder 6a and it mixes, a luminous layer 6 will be formed. 20a shows typically the conductive layer by the copper which originated in distortion and segregated here. This luminous layer 6 is arranged between the reflective insulating layer 5 on a back plate 4, and a transparent electrode 7. Through the moisture absorption films 2 and 2, these electroluminescence devices 1 are closed with the envelope films 3 and 3, and electroluminescence LGT 10 completes them. If high tension is impressed between two electrodes 4 and 7, the fluorescent substance 20 of a luminous layer 6 will emit light by two electrodes 4 and the electric field formed among seven. The electroluminescence LGT produced by the same approach as usual investigated brightness and a life. The alternating voltage of 1kHz–150V was impressed to these electroluminescence LGTs, the

brightness at that time was measured, and it considered as the brightness of each fluorescent substance. Moreover, the life impressed the same alternating voltage as the time of the measurement of luminance, and defined it by the time amount which initial brightness reduces by half. In addition, the life property was put in in the 50-degree C thermostat, and was performed in the state of the accelerated test. The obtained result is shown in the following table 1.

[0017]

[A table 1]

実施例	球の径 (mm)	球の比重 (g/cm <sup>3</sup> )	回転数 (rpm)	時間 (min)	輝度 (cd/m <sup>2</sup> )	寿命 (h)
1	5	3.91	1500	10	210	108
2	5	3.91	750	30	220	139
3	5	3.91	250	150	220	151
4	2	3.91	250	210	280	221
5	2	2.2	1500	10	243	143
6	2	2.2	250	210	258	152
7	1	5.7	250	210	298	213
8	1	3.91	250	210	290	245
9	1	2.2	250	210	257	178
10	0.75	3.91	1500	10	325	226
11	0.75	3.91	750	30	312	241
12	0.75	3.91	250	210	305	274
13	0.3	5.7	1500	10	259	155
14	0.3	5.7	750	30	261	143
15	0.3	5.7	250	210	254	140

[0018] In any case, brightness is 200 cd/m<sup>2</sup> so that clearly from a table 1. It exceeds and a life also exceeds 100 hours. At an example 12, they are 305 cd/m<sup>2</sup>. It is high and the longevity life on which a life also exceeds 270 hours is attained.

[0019] Except having used the diameter of sphere, as shown in the following table 2, the electroluminescence LGT was produced like the example and these brightness and lives were investigated. The obtained result is shown in a table 2.

[0020]

[A table 2]

実施例	球の径 (mm)	球の比重 (g/cm <sup>3</sup> )	回転数 (rpm)	時間 (min)	輝度 (cd/m <sup>2</sup> )	寿命 (h)
1	10	2.6	250	10	322	66
2	10	2.6	1500	30	314	63

[0021] Brightness is high so that clearly from a table 2, but since a fluorescent substance particle is ground and particle size becomes small, it is in the inclination for a life to become short.

[0022] Except having made small the specific gravity of three to example of comparison 4 ball, as shown in the following table 3, the electroluminescence LGT was produced like the example and these brightness and lives were investigated. The obtained result is shown in a table 3.

[0023]

[A table 3]

実施例	球の径 (mm)	球の比重 (g/cm <sup>3</sup> )	回転数 (rpm)	時間 (min)	輝度 (cd/m <sup>2</sup> )	寿命 (h)
3	1	1.5	250	240	100	312
4	1	1.5	1200	30	120	325

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[0024] When the specific gravity of a ball is made light so that clearly from a table 3, although a life becomes very long, it has the inclination for brightness not to be obtained.

[0025] Although the ball was used as a sphere in the above-mentioned example and the example of a comparison, an ellipsoid is sufficient, the thing of a potato-like indeterminate configuration may be used and, in short, there should just be no sharp edge section. Moreover, construction material can use general things, such as aluminum 2O3, ZrO2, SiAlON, and agate. In addition, a path shall be expressed as a diameter with a ball and things other than a ball shall be displayed with an overall diameter.

[0026] Moreover, others [ stirring / with the above-mentioned sphere as an approach of applying impulse force to a medium fluorescent substance / mixed ], How (this approach has the advantage that high brightness and an efficient fluorescent substance can be obtained more.) to contain a medium fluorescent substance independent or mixture with a sphere in a container, and to add an oscillation with predetermined acceleration and the amplitude How to contain in a container with flexibility and pressurize intermittently (this approach has the advantage that a longer lasting fluorescent substance can be obtained.) How (this approach has the advantage that a longer lasting fluorescent substance can be obtained.) to pass a medium fluorescent substance between the rollers of the rotating couple etc. — it is.

[0027] Thus, since a medium fluorescent substance particle has the high degree of hardness, the direction of impulse force instead of an isotropic pressure which concentrates locally and requires a pressure can produce distortion effectively in a medium fluorescent substance particle.

[0028]  
[Effect of the Invention] As explained above, by applying impulse force to a fluorescent substance and forming distortion in a fluorescent substance particle, copper segregates this invention to distortion and it can form a conductive layer. Moreover, since distortion can be formed effectively in a particle, without grinding a fluorescent substance particle by choosing as the suitable range the spherical diameter and specific gravity which give an impact, it is effective in the ability to obtain high brightness and the long lasting fluorescent substance for electroluminescence LGTs.

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**DESCRIPTION OF DRAWINGS**

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**[Brief Description of the Drawings]**

**[Drawing 1]** The important section expanded sectional view of the luminous layer which used the fluorescent substance manufactured by this invention.

**[Drawing 2]** The sectional view of the electroluminescence LGT concerning the former and this invention.

**[Drawing 3]** The important section expanded sectional view of the luminous layer which used the conventional fluorescent substance.

**[Description of Notations]**

- 1 Electroluminescence Devices
- 2 Moisture Absorption Film
- 3 Envelope Film
- 4 Back Plate
- 5 Reflective Insulating Layer
- 6 Luminous Layer
- 7 Transparent Electrode
- 8 Lead for Electrodes
- 9 Base Material Film for Transparent Electrodes
- 10 Electroluminescence LGT
- 6a An organic binder
- 6b Fluorescent substance
- 20 Fluorescent Substance Manufactured by this Invention
- 20a Conductive layer

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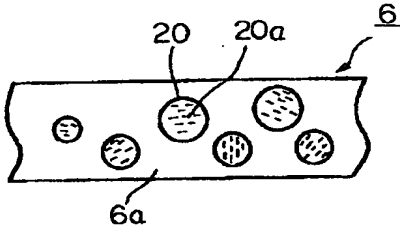
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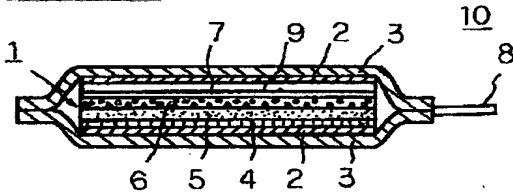
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## DRAWINGS

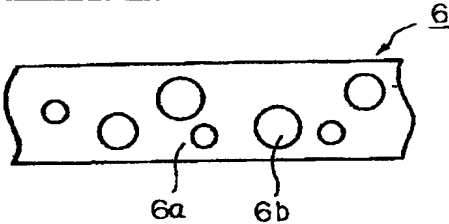
[Drawing 1]



[Drawing 2]



[Drawing 3]



[Translation done.]



(19) 日本国特許庁 (J P)

(12) 公開特許公報 (A)

(11) 特許出願公開番号

特開平6-306355

(43) 公開日 平成6年(1994)11月1日

(51) Int.Cl. <sup>5</sup>	識別記号	庁内整理番号	F I	技術表示箇所
C O 9 K 11/00		F 9159-4H		
11/08		B 9159-4H		
11/56	C P C	9159-4H		
H O 5 B 33/14				

審査請求 未請求 請求項の数3 O L (全 5 頁)

(21) 出願番号 特願平5-100579

(22) 出願日 平成5年(1993)4月27日

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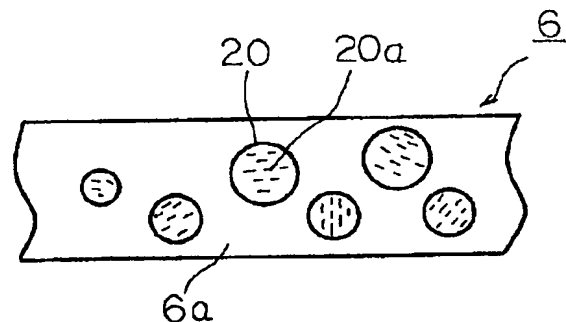
(54) 【発明の名称】 蛍光体の製造方法

(57) 【要約】

【目的】 高輝度・長寿命な電界発光灯用蛍光体を製造する。

【構成】 硫化亜鉛に付活剤として銅化合物を、共付活剤としてハロゲン化合物を添加し、その混合物を900～1100℃の温度で、1～5時間焼成する。次に、中間蛍光体に、0.1～5mm径で、2.0～6.0の比重の球を使用することにより、衝撃力を加えて、粒子内に歪みを生じさせる。そして、歪みを生じた中間蛍光体を、500～800℃の温度で、1～5時間焼成して、導電層20aを形成した蛍光体20を得る。

【効果】 蛍光体粒子内に積極的に歪みを導入することにより、蛍光体が活性化され、この蛍光体を使用すると高輝度、長寿命の電界発光灯を得ることができる。



## 【特許請求の範囲】

【請求項1】 硫化亜鉛に銅化合物とハロゲン化合物とを添加した混合物を比較的高温で焼成して中間蛍光体を製造する工程と、前記中間蛍光体に衝撃力を加えて蛍光体粒子内に歪みを生じさせる工程と、前記歪みを生じた中間蛍光体を前記焼成よりも低温で焼成して銅を偏析させる工程とを含むことを特徴とする蛍光体の製造方法。

【請求項2】 前記中間蛍光体粒子に衝撃力を加えて粒子内に歪みを生じさせる工程が中間蛍光体粒子と球体とを衝突させるものであって、該球体の径が0.1～5mmの範囲にあることを特徴とする請求項1記載の蛍光体の製造方法。

【請求項3】 前記球体の比重が、2.0～6.0の範囲になることを特徴とする請求項2記載の蛍光体の製造方法。

## 【発明の詳細な説明】

## 【0001】

【産業上の利用分野】 本発明は、蛍光体の製造方法に関し、特に電界発光蛍光体の製造方法に関する。

## 【0002】

【従来の技術】 電界発光灯は、文字や図形等の静止ディスプレイや液晶ディスプレイのバックライト等に利用されるもので、その構造を図2を参照して説明する。電界発光灯10は、偏平な矩形的電界発光素子1の表裏を吸湿フィルム2、2を介して、さらに2枚の外皮フィルム3、3でサンドイッチ的に挟んで封止したものである。電界発光素子1は、裏面側から表面側へ、アルミ箔からなる背面電極4、反射絶縁層5、蛍光体を含む発光層6、透明電極7を積層したもので、上記背面電極と透明電極の一辺からなる2本の電極引出し用のリード8、8が導出されている。上記発光層6は、図3に示すように、有機バインダ6a内に微小な球状の蛍光体6bを多数個分散して混入させたものである。上記リード8、8から背面電極4と透明電極7との間に高電圧を印加すると、両電極4、7間に形成される電界によって、両電極4、7間の発光層6の蛍光体6bが発光する。

【0003】 上記蛍光体6bは、一般的に粉体状の硫化亜鉛(ZnS)が母体として使用され、その母体に付活剤として銅(Cu)と、共付活剤としてハロゲン(例えばC1)を添加した混合物を焼成することにより得られる。

【0004】 この製造方法を説明する。硫化亜鉛100重量部に対して、0.02～0.1wt%の硫酸銅を添加してスラリー状にして混合し乾燥した後、この混合物に融剤として、アルカリ土類金属のハロゲン化合物およびアルカリ金属からなる群のうち少なくとも1種類を5～10wt%混合する。しかるのち、この混合物を石英るつぽに入れてふたをし、1100～1200℃の範囲の温度で3～10時間空気中で焼成し、焼成後脱イオン水で数回洗浄し、乾燥して中間蛍光体を作る。この中間蛍

光体に高圧をかけ、さらに、700～950℃でアニールすると、電界発光灯用蛍光体を製造することができる。(例えば、特開昭61-296085号公報)

【発明が解決しようとする課題】 ところで、上記の従来の電界発光灯用蛍光体の製造方法は、蛍光体の結晶型を転移させるために静水圧で加圧したもので、銅の偏析による導電層(Cu<sub>2</sub>Sといわれている)の形成が十分でなく、輝度、寿命が不足しているという欠点があった。

【0005】 本発明は、蛍光体粒子内に歪みを形成し該歪みに起因する導電層を形成することによって、高輝度、長寿命化を図った電界発光灯用蛍光体の製造方法を提供することを目的とする。

## 【0006】

【課題を解決するための手段】 上記目的を達成するため、本発明の蛍光体の製造方法は、硫化亜鉛に付活剤となる銅化合物と共付活剤となるハロゲン化合物とを添加した混合物を、比較的高温で焼成し、中間蛍光体を製造する工程と、前記中間蛍光体に衝撃力を加えて蛍光体粒子内に歪みを形成する工程と、前記歪みを生じた中間蛍光体を前記焼成よりも低温で焼成して銅を偏析させた蛍光体を製造する工程とを含むことを特徴とする。

【0007】 また、球体と中間蛍光体粒子の衝突により中間蛍光体に衝撃力を加えて粒子内に歪みを生じさせる工程において、球体に径が0.1～5mm、また、その比重が2.0～6.0の範囲にあることを特徴とする。

## 【0008】

【作用】 上記のような球体により、中間蛍光体粒子に衝撃力を加えると、中間蛍光体粒子を壊すほどの応力を与えずに、中間蛍光体粒子内に歪みを生じさせることが出来る。中間蛍光体粒子を壊さないで、EL特性を減じることなく、特に粒径が小さくならないので寿命特性に良い影響をもたらす。

【0009】 中間蛍光体粒子内に歪みを生じているので、比較的低温の焼成により、容易に銅の偏析を生じ、この偏析した銅が導電層として働くので輝度が大幅に向上する。

【0010】 中間蛍光体粒子は、硬いが脆い性質を持っている。したがって、静水圧のような等方的な圧力により、蛍光体粒子を加圧した場合、圧縮応力に強く、加圧を取り去ると元に戻り、蛍光体粒子に歪みを生じさせにくい。しかし、本発明のような球体と蛍光体粒子の衝突による応力では、異方的に圧力がかかるので、蛍光体粒子に歪みを生じさせることができる。

## 【0011】

【実施例】 以下、この発明を実施例に基づいて説明する。

【0012】 従来と同様、微粉末状の硫化亜鉛100重量部に対して、銅化合物例えば硫酸銅をCu濃度が0.05～0.2mol%となるように添加して純水と混合し、よく攪拌した後、この混合物をオープン内で加熱乾

燥する。その乾燥した混合物にフラックスとして、アルカリ土類金属のハロゲン化物およびアルカリ金属からなる群のうち少なくとも2種以上を総量5～25mol%混合する。

【0013】次に、この混合物をアルミナるつぽに入れてふたをし、900～1100℃の範囲の比較的高温で1～5時間空中で焼成する。その時、フラックスを介して微粉末状の硫化亜鉛が凝集して、10～40μmの粒子に成長する。その中間蛍光体と直径0.1～10mmの球とを容器にに入れて100～1500rpmの回転数で10～240分間攪拌し、中間蛍光体に衝撃力を加えた。この条件では、中間蛍光体粒子を割ったり、粉碎することなく、粒子内に歪みを形成することができる。

【0014】次に、その歪みを生じた中間蛍光体を、前記した焼成よりも比較的低温の500～800℃、1～5時間空中で焼成する。その時に、前記した焼成で中間蛍光体粒子内に均一拡散していた銅が、加熱されたことにより動き易くなり、粒子内の歪みのある場所に銅が偏析され、導電層が形成される。

【0015】その後、従来と同様、蛍光体表面にある不要物をKCN水溶液で除去した。KCN成分を水洗浄し\*

\*た後、オープン内で乾燥し、ふるい分けして蛍光体として完成する。

【0016】このようにして作製した蛍光体20を有機バインダ6a中に分散して混入すると、発光層6が形成される。ここで20aは歪みに起因して偏析した銅による導電層を模式的に示したものである。この発光層6を、背面電極4上の反射絶縁層5と、透明電極7との間に配置する。この電界発光素子1は、吸湿フィルム2、2を介して、外皮フィルム3、3で封止して電界発光灯10が完成する。両電極4、7間に高電圧を印加すると、両電極4、7間に形成される電界によって、発光層6の蛍光体20が発光する。従来と同様の方法で作製した電界発光灯により、輝度および寿命を調べた。これらの電界発光灯に1kHz～150Vの交流電圧を印加し、そのときの輝度を測定し、各蛍光体の輝度とした。また、寿命は、輝度測定時と同じ交流電圧を印加し、初期輝度が半減する時間により定義した。なお、寿命特性は50℃の恒温槽内に入れ、加速試験状態で行った。得られた結果を下記表1に示す。

【0017】

【表1】

実施例	球の径 (mm)	球の比重 (g/cm <sup>3</sup> )	回転数 (rpm)	時間 (min)	輝度 (cd/m <sup>2</sup> )	寿命 (h)
1	5	3.91	1500	10	210	108
2	5	3.91	750	30	220	139
3	5	3.91	250	150	220	151
4	2	3.91	250	210	280	221
5	2	2.2	1500	10	248	143
6	2	2.2	250	210	258	152
7	1	5.7	250	210	298	213
8	1	3.91	250	210	290	245
9	1	2.2	250	210	257	178
10	0.75	3.91	1500	10	325	226
11	0.75	3.91	750	30	312	241
12	0.75	3.91	250	210	305	274
13	0.3	5.7	1500	10	259	155
14	0.3	5.7	750	30	281	143
15	0.3	5.7	250	210	254	140

【0018】表1から明らかなように、いずれの場合も、輝度は200cd/m<sup>2</sup>を越え、寿命も100時間を越える。実施例12では305cd/m<sup>2</sup>と高く、寿命も270時間を越える長寿命が達成される。

【0019】球の直径を下記表2に示すように使用した

以外は、実施例と同様にして電界発光灯を作製し、これらの輝度および寿命を調べた。得られた結果を表2に示す。

【0020】

【表2】

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実施例	球の径 (mm)	球の比重 (g/cm <sup>3</sup> )	回転数 (rpm)	時間 (min)	輝度 (cd/m <sup>2</sup> )	寿命 (h)
1	10	2.6	250	10	322	66
2	10	2.6	1500	30	314	63

【0021】表2から明らかなように、輝度は高いが、蛍光体粒子が粉碎され粒径が小さくなるために、寿命が短くなる傾向にある。

【0022】比較例3～4

球の比重を下記表3に示すように小さくした以外は、実\*

\* 施例と同様にして電界発光灯を作製し、これらの輝度および寿命を調べた。得られた結果を表3に示す。

【0023】

10 【表3】

実施例	球の径 (mm)	球の比重 (g/cm <sup>3</sup> )	回転数 (rpm)	時間 (min)	輝度 (cd/m <sup>2</sup> )	寿命 (h)
3	1	1.5	250	240	100	312
4	1	1.5	1200	30	120	325

【0024】表3から明らかなように、球の比重を軽くすると、寿命は非常に長くなるが輝度が得られない傾向がある。

【0025】上記実施例、比較例では球体として球を使用した。が、楕円体でもよいし、じゃがいも状の不定形状のものでもよく、要はするどいエッジ部のないものであればよい。また、材質はAl<sub>2</sub>O<sub>3</sub>、ZrO<sub>2</sub>、SiAlON、メノウなど一般的なものが使用できる。なお、径は球では直径で表示し、球以外のものは最大径で表示するものとする。

【0026】また、中間蛍光体に衝撃力を加える方法として、上記の球体との混合攪拌のほか、中間蛍光体単独または球体との混合物を容器に収納して所定の加速度、振幅で振動を加える方法（この方法はより高輝度、高効率な蛍光体を得ることができるという利点がある。）、可撓性のある容器に収納して間欠的に加圧する方法（この方法はより長寿命な蛍光体を得ることができるという利点がある。）、回転する一對のローラー間に中間蛍光体を通過させる方法（この方法はより長寿命な蛍光体を得ることができるという利点がある。）などがある。

【0027】このように、中間蛍光体粒子は硬度が高いために、等方的な圧力ではなく、局部的に集中して圧力がかかるような衝撃力の方が中間蛍光体粒子内に有効に歪みを生じさせることができる。

【0028】

【発明の効果】以上説明したように、本発明は蛍光体に衝撃力を加えて蛍光体粒子内に歪みを形成することによ

り、銅が歪みに偏析し、導電層が形成できる。また、衝撃を与える球体の直径、比重を適当な範囲に選択することにより、蛍光体粒子を粉碎することなく、粒子内に有効に歪みを形成できるので、高輝度、長寿命の電界発光灯用蛍光体を得ることができるという効果がある。

【図面の簡単な説明】

【図1】 本発明により製造した蛍光体を使用した発光層の要部拡大断面図。

【図2】 従来および本発明に係わる電界発光灯の断面図。

【図3】 従来の蛍光体を使用した発光層の要部拡大断面図。

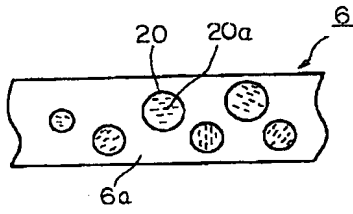
【符号の説明】

- 1 電界発光素子
- 2 吸湿フィルム
- 3 外皮フィルム
- 4 背面電極
- 5 反射絶縁層
- 6 発光層
- 7 透明電極
- 8 電極用リード
- 9 透明電極用基材フィルム
- 10 電界発光灯
- 6a 有機バインダ
- 6b 蛍光体
- 20 本発明により製造された蛍光体
- 20a 導電層

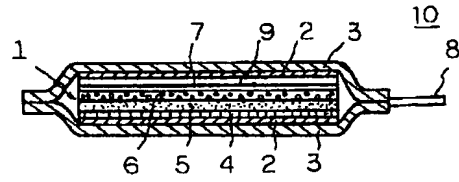
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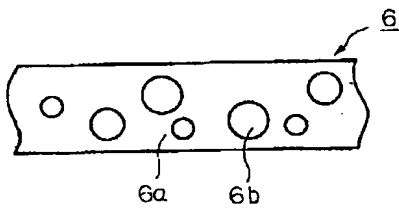
【図1】



【図2】



【図3】



### Concise explanation of documents

#### JP-A-6-306355

JP-A-6-306355 discloses phosphor powder of zinc sulfide having high luminescence due to a strain-introducing method. The strain-introducing method relates to a ball mill method widely used in the art. However, the particle size distribution of the phosphor powder of zinc sulfide prepared in Example in JP-A-6-306355 was wide (the particle size was in the range from 10  $\mu\text{m}$  to 40  $\mu\text{m}$ ), which is larger than the size of the phosphor particles in the electroluminescence device of the present invention. In addition, JP-A-6-306355 fails to disclose a preferred particle size and a variation coefficient thereof, a relationship between those and stacking faults, and a relationship between those and electroluminescence device structure.

#### JP-A-8-183954

JP-A-8-183954 discloses an electroluminescence phosphor, comprising a zinc sulfide as a host material thereof, wherein an particle diameter is 25  $\mu\text{m}$  or less, wherein an average face distance of stacking faults is 2 to 10 nm. However, JP-A-8-183954 fails to disclose a preferred particle size and a variation coefficient of

said preferable particle diameter distribution. Contrary to the above, these are disclosed in the present specification. Further, JP-A-8-183954 fails to disclose a preferred device structure using the same (e.g., a thickness of a phosphor layer) and the like.